



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Application No. : 10/615,926 Confirmation No. : 9914
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Filed : July 10, 2003
TC/A.U. : 1763
Examiner : J. R. Lund

Docket No. : 010986.52578US
Customer No. : 23911

Title : Film-Formation Apparatus and Source Supplying
Apparatus Therefor, Gas Concentration Measuring Method

**SUBMISSION OF ENGLISH-LANGUAGE TRANSLATION OF
PRIORITY DOCUMENT**

Commissioner for Patents
P.O. Box 1450
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Sir:

Applicant submits herewith an English-language translation of the priority document, JP 2002-201532, filed on July 10, 2002, for the above-referenced application, together with a Declaration of the translator indicating that the translation is accurate.

The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment, to Deposit Account No. 05-1323, Docket No.: 010986.52578US.

June 28, 2006

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I, Tadahiko Itoh, a Patent Attorney of Tokyo, Japan having my office at 32nd Floor, Yebisu Garden Place Tower, 20-3 Ebisu 4-Chome, Shibuya-Ku, Tokyo 150-6032, Japan do solemnly and sincerely declare that I am the translator of the attached English language translation and certify that the attached English language translation is a correct, true and faithful translation of Japanese Patent Application No. 2002-201532 to the best of my knowledge and belief.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

JUN. 26. 2006



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JPA No. 2002-201532

(Document Name)	Application for Patent
(Reference Number)	JPP020037
(Date of Submission)	July 10, 2002
(Destination)	Commissioner of Patent Office
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(IPC)	H01L 21/00
	G01N 21/35
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(Indication of Official Fees)	
(Prepayment Ledger Number)	002989
(Amount Paid)	¥21,000
(Lists of Submitted Documents)	
(Document Name)	Specification 1
(Document Name)	Drawing 1
(Document Name)	Abstract 1
(Proof Requested or Not)	Requested



-1-

[Name of the Document] Specification

[Title of the Invention]

FILM-FORMATION APPARATUS AND SOURCE SUPPLYING
APPARATUS THEREFOR

5 [Claims]

1. A film-formation apparatus, comprising:

a film-formation chamber; and

a source gas supplying apparatus supplying a
source gas to said film-formation chamber together with
10 a carrier gas,

said source gas supplying apparatus
comprising:

a concentration detector detecting a
concentration of said source gas; and

15 a gas flow controller controlling a flow rate
of an inert gas added to said carrier gas based on a
result of measurement of said concentration of said
source gas obtained by said concentration detector.

20 2. The film-formation apparatus as claimed in
claim 1, wherein said inert gas is added to said carrier
gas that is carrying said source gas.

25 3. The film-formation apparatus as claimed in
claim 1 or 2, wherein said concentration detector is
provided so as to measure said concentration of said
source gas in the state said inert gas is added to said
carrier gas.

4. The film-formation apparatus as claimed in any one of claims 1 to 3, wherein said gas flow controller changes said flow rate of said inert gas added to said carrier gas such that said concentration
5 of said source gas as measured by said concentration detector falls in a predetermined concentration range.

5. The film-formation apparatus as claimed in any one of claims 1 to 4, wherein said concentration
10 detector measures said concentration of said source gas before commencement of a film-formation process and/or during said film-formation process.

6. The film-formation apparatus as claimed in
15 any one of claims 1 to 5, wherein said source gas supplying apparatus further includes a switching device switching a flow path of said carrier gas added with said inert gas, between a first path connected to said film-formation chamber and a second path bypassing said
20 film-formation chamber, said concentration detector being provided in one of said first and second flow paths.

7. The film-formation apparatus as claimed in
25 any one of claims 1 to 6, wherein said gas flow controller changes a flow rate of said inert gas to be added to said carrier gas, and wherein said gas flow controller further changes a flow rate of said carrier gas such that a total flow rate of said carrier gas and
30 said inert gas is maintained generally constant.

8. The film-formation apparatus as claimed in any one of claims 1 to 7, wherein said carrier gas and said inert gas are introduced from an identical flow path, said inert gas being then diverted to another flow path before said carrier gas is admixed with said source gas, said inert gas merging again with said flow path of said carrier gas after said carrier gas is admixed with said inert gas.

10

9. The film-formation apparatus as claimed in any one of claims 1 to 8, wherein said gas flow controller controls a flow rate of said inert gas in said another flow path.

15

10. The film-formation apparatus as claimed in any one of claims 1 to 9, wherein said source gas is formed by vaporizing a source material of which vapor pressure less than 266Pa at a temperature at which said source material is used.

20

11. The film-formation apparatus as claimed in any one of claims 1 to 9, wherein said source gas is $W(CO)_6$.

25

12. The film-formation apparatus as claimed in any one of claims 1 to 11, wherein said concentration detector is a Fourier transform infrared spectrometer.

30

13. A source supplying system of the film-

formation apparatus as claimed in any one of claims 1 to 11.

[Detailed Description of the Invention]

5 [Field of the Invention]

The present invention generally relates to film-formation apparatuses, and more particularly to a CVD apparatus that can monitor and control the source gas concentration by way of an infrared spectrometer.

10 [Prior Art]

In the film-formation process according to CVD (chemical vapor deposition), particularly MOCVD (metal-organic chemical vapor deposition), in which formation of film is achieved by using an MO (metal-organic) source, a liquid source compound that contains the constituent elements of the film to be formed, or a liquid source prepared by dissolving a solid source compound containing such constituent elements into a solvent, is transported to a vaporizer located near to the processing vessel. There, the vaporizer causes vaporization of the source compound thus transported and there is produced a source gas as a result.

In the case of supplying a source gas from a bubbler to the processing vessel via a source gas line, the concentration of the source gas supplied to the processing vessel has been easily adjusted by merely controlling the carrier gas flow rate or pressure. Thus, there has been no particular need of direct detection

and monitoring of the source gas concentration supplied to the processing vessel in the conventional CVD technology.

In the case of forming high-K dielectric films or ferroelectric films, which are used in recent advanced semiconductor devices, or in the case of forming a tungsten (W) film or a ruthenium (Ru) film used also in these semiconductor devices, on the other hand, the vapor pressure of the source gas obtained from a source material is generally very low, and there can be a case in which the amount of the source gas sufficient for applying an ordinary source gas concentration control, which relies upon control of vaporization of the source gas, cannot be achieved by way of vaporization of the source material.

Thus, in the case of conducting a CVD process with such a low vapor pressure source material, a very small amount vapor produced by holding the source material at a predetermined temperature is supplied to the processing vessel as a source gas by using a carrier gas. Thereby, there occurs extensive dilution of the source gas by the carrier gas, and there arise a case in which accurate determination of the source gas concentration for the source gas actually introduced into the processing vessel is difficult.

Particularly, in the case of conducting a desired CVD process while using a solid source compound of low vapor pressure, there may occur a change of state of the source material with consumption of the source material. Such a change may occur, particularly with

regard to the effective surface area of the source material contacting with the carrier gas. When such a change of surface area is caused in the source material, it is generally not possible to avoid significant fluctuation of source gas concentration. Further, such a solid source material has a tendency of forming a temperature distribution inside because of poor heat conduction, contrary to the case of using a liquid source material. This also contributes to the tendency of deviation of the source gas concentration from the proper concentration range.

As noted above, it is preferable to measure or monitor the source gas concentration directly when conducting a CVD process when such a low-vapor pressure compound is used for the source material. On the other hand, the conventional gas concentration measurement method, such as the one that uses acoustic emission (AE) or specific heat, has a drawback in that reliable measurement is not possible when the measurement is conducted under a low-pressure environment such as the pressure of 10 Torr (1330 Pa) or less. Thus, such a conventional measurement process of gas concentration is not applicable to the case of the CVD film-formation process conducted while using a low-vapor pressure source material such as the MOCVD process.

Meanwhile, there is proposed a film-formation apparatus disclosed in the Japanese Laid-Open Patent Publication 2001-234348 that is capable of measuring the source gas concentration directly by Fourier-transform infrared (FTIR) spectrometer. This prior art film

formation apparatus also controls the gas flow rate based on the result of measurement of the source gas concentration. In this conventional film-formation apparatus and method, the mixing ratio of plural gas species is measured by FTIR, and the mixing ratio is adjusted by controlling the carrier gas flow rate ratio based on the result of the measurement.

[Problems to be Solved by the Invention]

Thus, in the case of using FTIR, it is possible to detect a concentration ratio of the source gases directly even in a low-pressure environment as used in an MOCVD process. On the other hand, it is not always easy to correct the concentration of the source gases in such a process when it was found, as a result of the FTIR measurement, that the source gas concentration ratio is deviated from a proper concentration range.

More specifically, the foregoing prior art film-formation process compensates for the change of source gas concentration by increasing or decreasing the carrier gas flow rate when it was judged by the FTIR measurement that the source gas concentration is deviated from a proper concentration range.

In such a control scheme, there is a possibility that increase or decrease of the carrier gas flow rate invites, depending on the vaporization rate of the liquid or solid source material used and also on the carrier gas flow rate, an unpredictable change of source gas concentration for the source gas that is actually

introduced into the processing vessel.

Consider now the case of increasing the carrier gas flow rate, under the situation that the source gas concentration in the carrier gas is judged as being smaller than the proper concentration range, such that the vaporization of the source material is facilitated and the concentration of the source gas supplied to the CVD film-formation chamber is increased as a result. There can be a case in which the vaporization of the source material cannot follow the increase of the carrier gas flow rate. When this is the case, the source gas produced from the source material is merely diluted by a large amount of the carrier gas, and the source gas concentration in the carrier gas is reduced, contrary to what is intended. Thereby, it becomes necessary to conduct a complex and time-consuming control process for recovering the desired source gas concentration.

In the case of decreasing the carrier gas flow rate under the situation in which the source gas concentration in the carrier gas is judged as being larger than the proper concentration range, so as to reduce the vaporization of the source material and decrease the concentration of the source gas introduced into the CVD chamber, the source gas vaporized from the source material is concentrated as a result of use of small amount of carrier gas. When this is the case, the source gas concentration in the carrier gas is increased, contrary to what is intended.

Further, while it is possible to control the

source gas concentration by adjusting the temperature of the liquid or solid source material, such a procedure is not realistic for the exact and quick control of the source gas concentration in view of the fact that the

5 vaporization rate changes drastically with the bottle temperature and in view of the fact that it requires a very rigorous temperature regulation for achieving the desired gas concentration. In addition, it should be noted that the response speed of the source gas

10 temperature is generally slow even when the vaporization temperature itself can be changed quickly and exactly during the film-formation process. Thus, the use of such a process of controlling the vaporization temperature is not realistic in the situation in which there is a

15 demand of quick and exact adjustment of the source gas concentration.

Accordingly, it is a general object of the present invention to provide a CVD apparatus as well as a source gas supplying apparatus used therefor, wherein

20 the concentration of a source gas supplied to a processing vessel of the CVD apparatus together with a carrier gas for conducting a CVD process is adjusted with high precision and with high speed even when the CVD process is conducted by using a low vapor pressure

25 source material.

[Means to Solve the Problems]

According to the present invention, as described in claim 1, there is provided a film-formation apparatus, comprising:

5 a film-formation chamber; and

a source gas supplying apparatus supplying a source gas to said film-formation chamber together with a carrier gas,

10 said source gas supplying apparatus comprising:

a concentration detector detecting a concentration of said source gas; and

15 a gas flow controller controlling a flow rate of an inert gas added to said carrier gas based on a result of measurement of said concentration of said source gas obtained by said concentration detector.

20 According to the present invention, as described in claim 2, there is provided a film-formation apparatus as claimed in claim 1, wherein said inert gas is added to said carrier gas that is carrying said source gas.

25 According to the present invention, as described in claim 3, there is provided a film-formation apparatus as claimed in claim 1 or 2, wherein said concentration detector is provided so as to measure said concentration of said source gas in the state said inert gas is added to said carrier gas.

According to the present invention, as described in claim 4, there is provided a film-formation

apparatus as claimed in any one of claims 1 to 3,
wherein said gas flow controller changes said flow rate
of said inert gas added to said carrier gas such that
said concentration of said source gas as measured by
5 said concentration detector falls in a predetermined
concentration range.

According to the present invention, as
described in claim 5, there is provided a film-formation
apparatus as claimed in any one of claims 1 to 4,
10 wherein said concentration detector measures said
concentration of said source gas before commencement of
a film-formation process and/or during said film-
formation process.

According to the present invention, as
15 described in claim 6, there is provided a film-formation
apparatus as claimed in any one of claims 1 to 5,
wherein said source gas supplying apparatus further
includes a switching device switching a flow path of
said carrier gas added with said inert gas, between a
20 first path connected to said film-formation chamber and
a second path bypassing said film-formation chamber,
said concentration detector being provided in one of
said first and second flow paths.

According to the present invention, as
25 described in claim 7, there is provided a film-formation
apparatus as claimed in any one of claims 1 to 6,
wherein said gas flow controller changes a flow rate of
said inert gas to be added to said carrier gas, and
wherein said gas flow controller further changes a flow
30 rate of said carrier gas such that a total flow rate of

said carrier gas and said inert gas is maintained generally constant.

According to the present invention, as described in claim 8, there is provided a film-formation apparatus as claimed in any one of claims 1 to 7, wherein said carrier gas and said inert gas are introduced from an identical flow path, said inert gas being then diverted to another flow path before said carrier gas is admixed with said source gas, said inert gas merging again with said flow path of said carrier gas after said carrier gas is admixed with said inert gas.

According to the present invention, as described in claim 9, there is provided a film-formation apparatus as claimed in any one of claims 1 to 8, wherein said gas flow controller controls a flow rate of said inert gas in said another flow path.

According to the present invention, as described in claim 2, there is provided a film-formation apparatus as claimed in any one of claims 1 to 9, wherein said source gas is formed by vaporizing a source material of which vapor pressure less than 266Pa at a temperature at which said source material is used.

According to the present invention, as described in claim 11, there is provided a film-formation apparatus as claimed in any one of claims 1 to 9, wherein said source gas is $W(CO)_6$.

According to the present invention, as described in claim 12, there is provided a film-formation apparatus as claimed in any one of claims 1 to

11, wherein said concentration detector is a Fourier transform infrared spectrometer.

According to the present invention, as described in claim 13, there is provided a source
5 supplying system of the film-formation apparatus as claimed in any one of claims 1 to 11.

According to the present invention, it becomes possible to supply the source gas always with a proper concentration at the time of film formation process, by
10 controlling the concentration of the source gas before or during the film-formation process. As a result, it becomes possible to conduct the film formation process with excellent film quality, reliability and reproducibility. As the source gas is adjusted to have
15 the proper concentration in the state the inert gas is added thereto, any deviation from the proper concentration can be immediately corrected by merely increasing the flow rate of the inert gas, as in the case of when it is judged that the measured
20 concentration of the source gas has exceeded the upper limit of the proper compositional range. In the event the measured concentration of the source gas has decreased below the lower limit of the proper concentration range, on the other hand, the proper
25 concentration is restored by merely decreasing the flow rate of the inert gas.

It should be noted that this inert gas functions as a diluting gas, and the present invention controls the flow rate of this diluting gas based on the
30 source gas concentration measured by the detector with

reference to the predetermined proper concentration range. This adjustment of the source gas concentration by way of control of the inert gas added to the source gas is predictable and can be achieved with high
5 precision and high speed, in contrast to the case of attempting such an adjustment by way of controlling the carrier gas flow rate.

It should be noted that the concentration of the source gas is measured in the state the inert gas is
10 added thereto in the present invention. In other words, the concentration of the source gas is measured in the state it is introduced into the CVD apparatus for film formation. By measuring the concentration of the source gas in the flow passage that is connected to the
15 processing chamber of a CVD apparatus, and particularly by measuring the concentration of the gas directly, a direct and reliable control of the source gas concentration becomes possible.

By using a Fourier transform infrared
20 spectrometer or a non-dispersion type infrared spectrometer, which shows high precision and high sensitivity also under a low-pressure environment as the means of the measurement of the source gas concentration, it becomes possible to control the film formation
25 process that uses a low vapor pressure source material such as a solid source material effectively. As noted before, there is a tendency that the source gas flow rate fluctuates significantly in the case a solid source material is used.

30 [Embodiments of the Invention]

[FIRST EMBODIMENT]

FIG.1 shows the construction of a processing vessel 100 used in a first embodiment of the present invention in a cross-sectional view.

5 Referring to FIG.1, the processing vessel 100 includes a processing vessel body 120 and a stage 130 provided in the processing vessel body 120 for supporting a semiconductor wafer 101, wherein the stage 130 is embedded with a heating element 132 driven by a power source 132A, and there is provided a shower head 110 inside the processing vessel body 120 so as to face the stage 130. The showerhead 110 introduces a gas supplied from a source gas line 30 into the process space inside the processing vessel body 120.

15 Further, there is provided a gate valve 140 at the sidewall of the processing vessel body 120 for loading and unloading the semiconductor wafer 101 to and from the processing vessel body 120. The processing vessel body 120 is evacuated via an evacuation line 32.

20 FIG.2 shows the construction of an MOCVD apparatus 200 that uses the processing vessel 100 of FIG.1 schematically.

Referring to FIG.2, the MOCVD apparatus 200 includes a source bottle 10, wherein the source bottle 10 is supplied with an inert gas such as Ar, Kr, N₂, H₂, and the like, from a source gas line 30 via a mass flow controller (MFC) 12A, which is provided in a part of the source gas line 30. Thereby, the mass-flow controller 12A controls the flow rate of the inert gas supplied to the source bottle 10.

The source bottle 10 accommodates therein a liquid or solid source material and produces a source gas therein as a result of vaporization of the source material. The inert gas thus supplied to the source
5 bottle 10 functions as a carrier gas and transports the source gas from the source bottle 10 to the processing vessel 100. Thereby, the source gas flows out from the source bottle 10 at the outlet port thereof and is transported along the source gas line 30. It should be
10 noted that there is provided a manometer 18 in the vicinity of the outlet port of the source bottle 10 for detecting the pressure inside the source bottle 10.

In the MOCVD apparatus 200 of FIG.14, it should further be noted that there is provided a
15 diluting gas line 31 to the source gas line 30 so as to merge at a downstream side of the manometer 18, and an inert gas such as Ar, Kr, N₂, H₂, and the like, is supplied to the diluting gas line 31 via another mass-flow controller 12B. It should be noted that the mass-
20 flow controller 12B controls the flow rate of the inert gas added to the source gas line 30.

It should be noted that this inert gas in the diluting gas line 31 functions as a diluting gas when it is added to the source gas line 30 and dilutes the
25 source gas transported through the source gas line 30 from the source bottle 10. Hereinafter, the gas in the source gas line 30 thus diluted by the inert gas in the gas line 31 will be referred to as a "mixed gas". This mixed gas is supplied to the processing vessel 100
30 through the source gas line 30.

In the construction of FIG.2, a turbo molecular pump (TMP) 14 is provided to the evacuation line 32 connected to the processing vessel 100, and there is further provided a dry pump (DP) behind the turbo molecular pump 14 for boosting the same. By driving these pumps 14 and 16, the interior of the processing vessel body 120 is maintained at a predetermined pressure. For example, the turbo molecular pump 14 can evacuate the process space inside the processing vessel 120 to a high-degree vacuum state characterized by the pressure of about 1 Torr (133Pa). Thereby, it becomes possible to conduct the film formation process that uses a source material of low vapor pressure.

It should be noted that there is provided a pre-flow line 33 in the source gas line 30 so as to bypass the processing vessel 100 at the downstream side of the source bottle 10, and the mixed gas in the source gas line 30 is supplied selectively to one of the pre-flow line 33 or to the source gas line 30 connected to the processing vessel 100 by the switching of a valve 26 provided on the source gas line 30 from the source bottle 10. The pre-flow line 33 is provided for stabilizing the flow rate of the mixed gas supplied to the processing vessel 100 at the time of the film-formation process and to pre-adjust the concentration of the mixed gas. Thus, the mixed gas is caused to flow through the pre-flow line 33 in advance to each step of processing the substrate 101.

Meanwhile, the mixed gas supplied to the

processing vessel 100 has to contain the source gas with a proper concentration range in order to achieve the desired film formation. Further, the mixed gas is required to contain the source gas with a constant
5 concentration within the proper concentration range in order to avoid variation of film quality in each film-formation step when the film-formation step is conducted repeatedly.

In the MOCVD apparatus 200 of FIG.2, on the
10 other hand, there arises a difficulty of detecting the source gas concentration accurately because of the fact that the carrier gas and also the diluting gas are added to the source gas when supplying the source gas to the processing vessel 100, contrary to the case of supplying
15 the source gas directly from a vaporizer. As noted previously, the concentration of the source gas tends to change with the pressure variation or variation of the surface area of the source material, particularly when a solid source material is used, and it is difficult
20 stabilize the concentration of the source gas in the mixed gas.

Thus, according to a first embodiment of the present invention, variation of the source gas concentration in the mixed gas is detected with high
25 precision by using a Fourier-transform infrared spectrometer, and the flow rate of the inert gas is controlled by using the mass-flow controller 12B and/or 12A such that the source gas concentration in the mixed gas falls always in a predetermined, proper
30 concentration range.

More specifically, the present embodiment provides a Fourier-transform infrared spectrometer 40 referred to hereinafter as FTIR 40 in the pre-flow line 33 of the MOCVD apparatus 200, wherein the FTIR 40 includes a wave monitor using a laser light and a movable mirror. More specifically, the FTIR 40 includes an interferometer, infrared detector and a processing unit and measures the concentration of gas species contained in a gas based upon the absorption spectrum of respective gas species, by irradiating an infrared beam upon the gas via an interferometer and processing the output of the infrared detector by the processing unit.

The pre-flow line 33 is merged to the evacuation line 32 at the upstream side of the dry pump 16 and is set to the predetermined degree of vacuum by the dry pump 16. In the MOCVD apparatus 200 of FIG.2, it becomes possible to measure the source gas concentration under the pressure of 50 Torr (6660Pa) or less, in which concentration measurement by way of acoustic emission process is not possible, by providing the FTIR 40 in the pre-flow line 33.

In the present embodiment, it should be noted that the FTIR 40 provided in the pre-flow line 33 measures the source gas concentration in the mixed gas (referred to hereinafter as "measured concentration") and supplies a signal indicative of the measured concentration to a controller 201.

Thus, in the event it is judged in the controller 201 that the measured concentration change has exceeded a predetermined range, the controller 201

controls the mass-flow controller 12B and/or 12A, and increases or decreases the flow rate of the inert gas. It should be noted that this controller 201 may be provided inside the FTIR 40 or inside any of the mass-flow controller 12B and/or 12A.

According to the first embodiment of the present invention, the source gas concentration in the mixed gas is controlled constant before conducting a film formation step for each substrate, and the source gas is introduced into the processing vessel with controlled concentration in each film-formation process. Thereby, wafer-to-wafer variation of film quality is successfully minimized.

Because the source gas concentration is adjusted to the proper concentration range at the beginning of the process by adding the diluting gas to the carrier gas, it is possible to increase the source gas concentration immediately, in the event the source gas concentration has decreased during the film-formation process as a result of decrease of the source material or as a result of decrease of vaporization efficiency of the source material, and the like, by decreasing the flow rate of the diluting gas. Similarly, it is possible to decrease the source gas concentration immediately in the event the source gas concentration has increased during the film-formation process, by increasing the flow rate of the diluting gas.

As explained before, increase of the carrier gas flow rate does not always lead to increase of source gas concentration. This is particularly true in the case

of using a solid source material, in which there is caused a reduction of surface area with the progress of the film-formation process. In such a case, the efficiency of vaporization decreases when the carrier
5 gas flow rate is increased.

In the present invention, on the other hand, it is possible to increase the source gas concentration by merely decreasing the flow rate of the diluting gas. Of course, it is possible to change the carrier gas flow
10 rate simultaneously to the change of the diluting gas flow rate.

As the diluting gas does not contain the source gas, it is easy to determine the amount of the flow rate change from the measured concentration
15 obtained by FTIR and from the predetermined proper concentration range. Thus, the concentration control of the source gas by the increase or decrease of the diluting gas can be conducted at high speed with high precision, contrary to the case of conducting the
20 concentration control by way of control of the carrier gas alone.

[SECOND EMBODIMENT]

FIG.3 shows the construction of an MOCVD
25 apparatus 200A according to a second embodiment of the present invention, wherein those parts corresponding to the parts described previously are designated by the same reference numerals and the description thereof will be omitted.

30 Referring to FIG.3, it can be seen that the

inert gas such as Ar, Kr, N₂, H₂, and the like, is supplied to the source bottle 10 via the source gas line 30 via the mass-flow controller 12A, wherein the mass-flow controller 12A controls the flow rate of the inert gas to be supplied to the source bottle 10. The source bottle 10 accommodates therein a liquid or solid source used for the film-formation process. Thereby, the source gas is produced as a result of the vaporization of the source material in the source bottle 10. The inert gas thus supplied to the source bottle 10 is forwarded to the source gas line 30 from the outlet port of the source bottle 10 as the carrier gas.

According to the second embodiment of the present invention, it can be seen that the diluting gas line 31 is provided to the source gas line 30 at the downstream side of the mass-flow controller 12A so as to bypass the source bottle 10, and the diluting gas line 31 is provided with the inert gas diverted from the source gas line 30. This inert gas is admixed to the carrier gas carrying the source gas from the source bottle 10 upon merging to the source gas line at the node B as the diluting gas, and the mixed gas formed of the source gas, carrier gas and the diluting gas is caused to flow through the source gas line 30 at the downstream side of the node B. Thereby, the source gas in the mixed gas is diluted by the inert diluting gas in the gas line 31. The flow rate of this diluting gas is controlled by a valve 20 provided in the gas line 31.

This mixed gas is then supplied selectively to one of the processing vessel 100 or the pre-flow line 33

in which the FTIR 40 is provided, after passing through the source gas line 30.

The FTIR 40 of the pre-flow line 33 measures the source concentration in the mixed gas and supplies
5 an output signal indicative of the measured concentration to the controller 201. Thus, the controller 201 judges whether the measured concentration falls within the predetermined proper concentration range or not and conducts the control of increasing or
10 decreasing the diluting gas flow rate by controlling the valve 20 when it is judged that the measured concentration deviated beyond the proper concentration range.

According to the second embodiment of the
15 present invention, it becomes possible to minimize the wafer-to-wafer variation of the film quality by monitoring the source gas concentration by using the FTIR 40 provided in the pre-flow line 33, similarly to the first embodiment. Further, the present embodiment,
20 which uses FTIR for the concentration measurement, is suitable for the film-formation process that uses the source material of low vapor pressure. Further, any deviation of the source gas concentration in the mixed gas beyond the proper concentration range is corrected
25 immediately by increasing or decreasing the flow rate of the diluting gas.

In the present embodiment in which the flow rate of the diluting gas is controlled by the valve 20 provided in the diluting gas line 31, it is possible to
30 adjust the flow rate of the diluting gas and the carrier

gas by using the single mass-flow controller 12A. Further, the construction of the present embodiment, in which the diluting gas line 31 is branched from the line 30 and merged thereto again, has the feature that the inert gas flow rate before branching becomes generally equal to the inert gas flow rate at the merging node B. Thus, it becomes possible to maintain a constant flow rate for the mixed gas supplied to the processing vessel 100 while controlling the source gas concentration therein by way of increasing or decreasing the diluting gas flow rate. As a result, wafer-to-wafer variation of film quality is reduced further by using the present embodiment.

[THIRD EMBODIMENT]

FIG.4 shows the construction of an MOCVD apparatus 200B according to a third embodiment of the present invention, wherein those parts corresponding to the parts described previously are designated by the same reference numerals and the description thereof will be omitted.

Referring to FIG.4, it can be seen that the inert gas such as Ar, Kr, N₂, H₂, and the like, is supplied to the source bottle 10 from the mass-flow controller 12A through the source gas line 30, wherein the mass-flow controller 12A controls the flow rate of the inert gas supplied to the source bottle 10. The source bottle 10 accommodates therein a liquid or solid source material used for film-formation, and the source material undergoes vaporization in the source bottle 10.

The inert gas thus supplied to the source bottle 10 functions as a carrier gas and carries the source gas therewith. Further, the manometer 18 is provided in the vicinity of the gas outlet of the source bottle 10 for
5 detecting the pressure in the source bottle 10.

The source gas line 30 is further provided with the diluting gas line 31 such that the diluting gas line 31 merges at the downstream side of the manometer 18, and the diluting gas line 31 is supplied with an
10 inert gas such as Ar, Kr, N₂, H₂, and the like, via the mass-flow controller 12B. Thereby, mass-flow controller 12B controls the flow rate of the inert gas to be merged to the source gas line 30. It should be noted that this inert gas is admixed to the source gas and the carrier
15 gas from the source bottle 10 upon merging to the source gas line 30, and the mixed gas is formed in the source gas line 30 as a result of the admixing. Thereby, the inert gas thus added causes dilution of the source gas contained in the mixed gas. The mixed gas thus formed is
20 then supplied to the processing vessel 100 through the source gas line 30. In the present embodiment, it is also possible to construct the diluting gas line 31 similar to the one explained with reference to the second embodiment.

25 In the present embodiment, the evacuation line 32 for evacuating the processing vessel 100 is provided with the turbo molecular pump 14, and the turbo molecular pump 14 is boosted by the dry pump 16 provided at the downstream side of the turbo molecular pump 14.
30 Thereby, the process space in the processing vessel 100

is maintained at a predetermined pressure or predetermined degree of vacuum such as 1 Torr (133Pa) or less. Such a low pressure environment is particularly important in the film-formation process that uses a low
5 vapor pressure source material.

It should be noted that the source gas line 30 is provided with the pre-flow line 33 such that the pre-flow line 22 bypasses the processing vessel 100, wherein the pre-flow line 33 is supplied with the mixed gas in
10 the source gas line 30. Thereby, the mixed gas is supplied selectively to one of the pre-flow line 33 and the source gas line 30 connected to the processing vessel 100 by the activation of the valve 26. Here, the pre-flow line 33 is provided for stabilizing the flow
15 rage of the mixed gas supplied to the processing vessel 100 at the time of the film-formation process and to adjust the concentration of the mixed gas in advance. As can be seen, this pre-flow line 33 merges to the evacuation line 32 at the upstream side of the dry pump
20 16. Thus, the pressure inside the pre-flow line 33 is determined by the dry pump 16.

In the first and second embodiments described before, it should be noted that the concentration of the mixed gas supplied to the processing vessel 100 is
25 monitored by the FTIR 40 provided in the pre-flow line 33. In the case of switching the mixed gas hitherto supplied to the pre-flow line 33, to the source gas line 30 connected to the processing vessel in the gas supply system having such a construction, there can be a case
30 that the source gas concentration undergoes a change

before and after the switching as a result of different diameter of the gas lines or existence or non-existence of the processing vessel body 120 in the evacuation path, which causes a change of impedance in the evacuation system, and such a change of impedance of the evacuation system can cause a change of pressure in the source bottle 10. In the case of conducting film-formation by using a low vapor pressure source, in particular, the pressure inside the source bottle 10 is held at the pressure of 1 Torr (133Pa) or less because of the use of the turbo molecular pump 14 for facilitating the vaporization of the source material in the source bottle 10. On the other hand, the pre-flow line 33 is evacuated by the dry pump 16 alone, and thus, it is difficult to realize such a low pressure in the pre-flow line 33.

Even in such a case, the first and second embodiments can successfully reduce the wafer-to-wafer variation of film quality by maintaining the source gas concentration in the mixed gas flowing through the pre-flow line 22 before each film-formation step.

However, it would be more advantageous when a direct control of the source gas concentration in the mixed gas supplied actually to the processing vessel is achieved. Thereby, it should be noted that such a direct control of the source gas concentration in the mixed gas actually supplied to the processing vessel 100 has to be conducted in a short time period in view of the fact that the film-formation process is in progress in the processing vessel 100 while using this mixed gas.

The present invention can immediately correct

any deviation of the source gas concentration by way of increase or decrease of the diluting gas, and thus, it becomes possible to control the source gas concentration in the mixed gas supplied directly to the processing vessel 100.

More specifically, the present embodiment provides the FTIR 40 in the source gas line 30 connected to the processing vessel 100 for measuring the source gas concentration of the mixed gas introduced into the processing vessel 100. It should be noted that this source gas line 30 may be held at a low pressure such as 1 Torr (133Pa) or less by using the turbo molecular pump 14 and the dry pump 16 in order to facilitate vaporization of the low vapor pressure source material. Even in such a case, the source gas concentration is measured accurately by using the FTIR 40.

In the present embodiment, the FTIR 40 provided in the source supply line 30 connected to the processing vessel 100 measures the source gas concentration in the mixed gas and supplied an output signal indicative of the measured concentration to the controller 201. When the controller 201 judges that the measured concentration of the FTIR 40 has exceeded the proper concentration range, the controller 201 controls the mass-flow controller 12B and/or 12A and the flow rate of the inert gas added to the mixed gas is increased or decreased.

According to the fourth embodiment of the present invention, it becomes possible to immediately correct any deviation of the source gas concentration in

the mixed gas from the proper concentration range by increasing or decreasing the diluting gas flow rate similarly to the previous embodiments.

Thus, the present embodiment enables direct
5 measurement of the source gas concentration in the mixed gas, which is actually used for film formation, by using the FTIR 40 provided in the source gas line 40 connected to the processing vessel 100. Thereby, any deviation of the source gas concentration in the mixed gas currently
10 in use in an on-going film formation process is detected and corrected immediately. Thereby, the film-formation processing can be conducted by using a source gas of which concentration is always controlled to the proper compositional range, and the desired film quality is
15 maintained over the repeatedly conducted wafer-to-wafer film-formation processes.

[FOURTH EMBODIMENT]

FIG.5 shows the construction of an MOCVD
20 apparatus 200C according to a fourth embodiment of the present invention, wherein those parts corresponding to the parts described previously are designated by the same reference numerals and the description thereof will be omitted.

25 Referring to FIG.5, the inert gas such as Ar, Kr, N₂, H₂, and the like, is supplied to the source bottle 10 from the mass-flow controller 12A through the source gas line 30, wherein the mass-flow controller 12A controls the flow rate of the inert gas supplied to the
30 source bottle 10. The source bottle 10 accommodates

therein a liquid or solid source material used for the film-formation process, and the source gas is produced as a result of vaporization conducted in the source bottle 10. Thereby, the inert gas supplied to the source
5 bottle 10 functions as a carrier gas and carries the vaporized source gas therewith. Further, the manometer 18 is provided in the vicinity of the outlet port of the source bottle 10 connected to the source gas line 30 for detecting the pressure inside the source bottle 10.

10 Further, there is provided the diluting gas line 31 such that the diluting gas line 31 merges the source gas line 30 at the downstream side of the manometer 18, and the inert gas such as Ar, Kr, N₂, H₂, and the like, is supplied to the diluting gas line 31
15 via the mass-flow controller 12B. Thereby, the mass-flow controller 12B controls the flow rate of the inert gas added to the source gas supply line 30. It should be noted that this inert gas is added, upon merging to the source gas line 30, to the source gas and the carrier
20 gas from the source bottle 10 as a diluting gas, and there is formed a mixed gas in the source gas line 30 as a result of the mixing of the source gas, carrier gas and the diluting gas at the downstream side of the node where the gas line 31 merges the gas line 30. This mixed
25 gas is then supplied to the processing vessel 100 along the source gas supply line 30. Here, it is possible to construct the diluting gas line 31 and the source gas supply line 30 similarly to the second embodiment.

The source gas supply line 30 is provided with
30 the pre-flow line 33 so as to bypass the processing

vessel 100 at the downstream side of the source bottle 10, and the pre-flow line 33 is supplied with the mixed gas from the source gas line 30. The mixed gas is thereby supplied selectively to one of the pre-flow line 5 33 and the source gas line 30 connected to the processing vessel 100 by the activation of the valve 26. It should be noted that this pre-flow line 33 is provided so as to stabilize the flow rate of the mixed gas supplied to the processing vessel 100 at the time of 10 the film formation process and further to adjust the mixed gas concentration in advance. The pre-flow line 33 is connected to the evacuation line 32 at the upstream side of the dry pump 16. Thus, the pre-flow line 33 is evacuated to a predetermined pressure or degree of 15 vacuum by the dry pump 16.

According to the fourth embodiment of the present invention, the FTIR 40 is provided at the downstream side of the node where the gas line 31 merges the gas line 30 but at the upstream side of the node 20 where the pre-flow line 33 branches from the gas line 30, so as to enable measurement of the source gas concentration in the mixed gas introduced into the processing vessel 100 and so as to enable measurement of the source gas concentration in the mixed gas flowing 25 through the pre-flow line 33.

As shown in FIG.5, the FTIR 40 may be provided in a bypass line 35 bypassing from the source gas line 30. The bypass line 35 is provided with valves 21 and 25, and there is further provided a valve 23 in the source 30 gas line 30 between the node where the bypass line 35

branches from the source gas line 30 and the node where the bypass line 35 merges the source gas line 30.

As a result of opening and closing of these valves 21, 23 and 25, the mixed gas is supplied selectively to either the bypass line 35 or the source gas line 30. Thus, the mixed gas is supplied to the bypass line 35 the source gas concentration is to be measured. When there is no need of measurement of the source gas concentration, the source gas is supplied directly to the downstream part of the source gas line 30.

The output of the FTIR 40 is supplied to the controller 201, and the controller 201 controls the mass-flow controller 12A and/or 12B in response to the output signal of the FTIR 40.

According to the fourth embodiment described above, it is possible to correct any deviation of the source gas concentration in the mixed gas beyond a predetermined proper concentration range immediately by increasing or decreasing the flow rate of the diluting gas.

As the FTIR 40 is disposed so as to measure both the source gas concentration introduced into the processing vessel and the source gas concentration in the pre-flow line 33, the present embodiment can control not only the source gas concentration before the commencement of the film-formation process but also the source gas concentration actually used for the film-formation process. In other words, the present embodiment enables monitoring of the source gas

concentration of the mixed gas actually in use for the film formation process and correction of any deviation of the source gas concentration beyond the predetermined concentration range immediately. As the mixed gas used
5 for the actual film-formation process has the source gas concentration already adjusted during the period in which the mixed gas is caused to flow through the pre-flow line, there can occur no large deviation in the source gas concentration when the mixed gas is switched
10 and introduced into the processing vessel 100. Thus, the need of large change of the source gas concentration during the film-formation process by significantly increasing or decreasing the diluting gas is avoided, and a stable film formation becomes possible.

15 While the present invention has been described for the case of conducting film-formation process by using a single gas source specie, the present invention is applicable also to the case of conducting film-formation processing by using two or more gas species.
20 In such a case, two or more source gas lines are provided for supplying the respective source gases to the CVD apparatus. Otherwise, the CVD apparatus has a construction similar to those described before.

It should be noted that the pre-flow line 33
25 is indicated as merging to the evacuation line 32 at the upstream side of the dry pump 16. In such a case, there is a possibility that the source gas concentration is measured in the state in which the pressure of the mixed gas is higher than the case in which the mixed gas
30 actually flows to the processing vessel 100. In order to

avoid this problem and to measure the source gas concentration at the pressure in which the mixed gas is actually supplied to the processing vessel 100, it is possible to increase the diameter of the pipe of the pre-flow line 33 for the part extending from the outlet of the FTIR to the node where the pre-flow line 33 merges the evacuation line 32 at the upstream side of the dry pump 16. Alternatively, it is possible to merge the pre-flow line 33 to the evacuation line 32 not at the upstream side of the dry pump 16 but at the upstream side of the turbo molecular pump 14. Thereby, it is possible to provide a pressure regulation valve not illustrated in the pre-flow line 33 at the upstream side of the node where the pre-flow line 33 merges the evacuation line 32 and control the cell pressure to the pressure of the film-formation process when the FTIR 40 is activated to conduct the measurement of the source gas concentration.

Next, the control operation of the controller 201 in the various embodiments described heretofore will be explained.

FIG.6 shows an embodiment of control routine for controlling the source gas concentration in the mixed gas. While the illustration is omitted, this controller 201 is formed of a microcomputer including a CPU as a major component and stores the target source gas concentration $C1$ of the mixed gas, initial designed flow rate value $Q1$ of the diluting gas, the initial designed flow rate value $Q2$ of the carrier gas, and the like, in a memory.

In the step S300, the microcomputer sets the flow rate of the diluting gas to the value Q1 based on the data stored in the memory and produces a control signal setting the carrier gas flow rate to the value Q2.
5 This control signal is then supplied to the mass-flow controllers 12A and 12B.

Next, in the step 302, the microcomputer determines the flow rate of the diluting gas and the flow rate of the carrier gas in response to the measured
10 concentration C2 supplied from the FTIR 40 and determines the flow rate of the diluting gas and the flow rate of the carrier gas such that the measured concentration C2 coincides with the target concentration C1. Alternatively, the microcomputer decides whether or
15 not the measured concentration C2 is deviated from the target concentration C1 beyond the allowable range. In this case, the microcomputer determines the flow rate of the diluting gas and the flow rate of the carrier gas such that the measured concentration C2 falls in the
20 allowable range only when it is judges that the measured concentration C2 has deviated beyond the foregoing allowable range.

In the present embodiment, the overall flow rate of the diluting gas and the carrier gas is set
25 constant before and after the adjustment. Thus, the flow rate of the diluting gas after the adjustment can be represented as $Q1'=Q1+\beta$ and the flow rate of the carrier gas after the adjustment can be represented as $Q2'=Q2-\beta$. In the adjustment, the parameter β is
30 determined.

Thus, in the initial routine step 302, the term $-Q2/10$ or $+Q2/10$ is substituted into the parameter β and the initial values $Q1$ and $Q2$ are updated to $Q1'$ and $Q2'$. The values thus updated are stored in the memory, and the corresponding control signals are transmitted to the mass-flow controllers 12A and 12B. Further, the step 302 is repeated in response to the input from the FTIR 40.

In the step 302 of the next routine, a new parameter β is determined in such a manner that the difference between the newly measured concentration $C2$ and the target concentration $C1$ is decreased and such that the newly determined parameter β has a smaller magnitude. By using the newly determined parameter β , the initial values $Q1$ and $Q2$ are updated to $Q1'$ and $Q2'$ and stored in the memory. Thereby, the corresponding control signals are transmitted to the mass-flow controllers 12A and 12B.

[EXAMPLE 1]

FIG.7 shows an example of the infrared absorption spectrum of the metal organic gas $W(CO)_6$ (hexacarbonyl tungsten), wherein the horizontal axis represents the wavenumber while the vertical axis represents the transmissivity.

From FIG.7, it can be seen that there exists characteristic absorption of carbonyl group ($=CO$) in the metal organic gas $W(CO)_6$ at the wavenumbers of about 2900cm^{-1} , 1900cm^{-1} and 500cm^{-1} .

In order to confirm the sensitivity of the

FTIR 40 for the concentration change of the $W(CO)_6$ gas, an experiment was made in which the source bottle 10 is held at the temperatures of 25°C, 45°C and 60°C and an Ar carrier gas is supplied with the flow rate of 50 SCCM. Thereby, the FTIR 40 was provided not in the pre-flow line 33 but at the upstream side of the processing vessel 100 as in the case of the third embodiment. Here, the pressure of the FTIR cell becomes 80Pa, 85Pa and 87Pa, respectively, and it was found that the corrected absorbance of the carbonyl group corrected to the pressure of 1330Pa (10 Torr) is 0.337, 0.656 and 1.050, respectively. From this result, it was confirmed that the FTIR has a sufficient sensitivity even at such low pressures and that it can be used for the monitoring of the concentration change of the $W(CO)_6$ gas by monitoring the change of the absorption peak intensity.

[EXAMPLE 2]

In Example 2, $W(CO)_6$ is used for the source material and the source bottle 10 is held at 45°C. Further, an Ar gas was used for the carrier gas and the diluting gas. The FTIR 40 was provided at the upstream side of the processing vessel 100, similarly to the fourth embodiment, and the temperature of the source bottle was set to 45°C. Thereby, the carrier gas was supplied with the flow rate of 50 SCCM and the diluting gas was supplied with the flow rate of 10 SCCM.

In this experiment, a value of 0.235 was obtained initially for the absorbance of $W(CO)_6$ as corrected to the pressure of 1330Pa (10Torr) from the

absorption peak of the carbonyl group.

After 5 minutes, it was found that the value of the absorbance has changed to 0.267, and thus, the flow rate of the diluting gas was increased slightly to the value of 12SCCM. With this, it became possible to change the absorbance to 0.233, which is close to the original value.

[EXAMPLE 3]

10 In Example 3, a tungsten film was formed by a pyrolytic CVD process while using $W(CO)_6$ as a source material.

In this experiment, the source bottle 10 was held at $60^{\circ}C$ and an Ar carrier gas was supplied with the flow rate of 300SCCM. Further, an Ar diluting gas was supplied with the flow rate of 100SCCM.

Further, in order to facilitate the vaporization of $W(CO)_6$, which provides only the vapor pressure of about 106Pa at $60^{\circ}C$, and to increase the film-formation rate, the present embodiment activates the turbo molecular pump 14 and the dry pump 16 such that a process pressure of 0.15 Torr (about 20Pa) is realized in the processing vessel body 120 and the pressure of 1.5 Torr (about 200Pa) is realized in the source gas line 30.

As a result of the film formation conducted at the substrate temperature of $450^{\circ}C$, it was confirmed that there occurs a tungsten film formation with the rate of 7.1nm/min. The tungsten film thus formed had the resistivity of $27\mu\Omega cm$.

The present invention is not limited to the embodiments described heretofore, but various variations and modifications may be made without departing from the scope of the invention. For example, it is possible to
5 provide a turbo molecular pump in the pre-flow line 33 in correspondence to the film-formation process that uses a low vapor pressure source material. Further, the diameter of the pre-flow line 33 may be optimized. According to such a construction, it becomes possible to
10 measure the source gas concentration under the condition close to the case of the source gas is actually transported along the source gas line 30 for the film-formation process by using the FTIR 40 provided in the pre-flow line 33.

15 [Advantages of the Invention]

According to the present invention, it becomes possible to supply the source gas always with a proper concentration at the time of film formation process, by controlling the concentration of the source gas before
20 or during the film-formation process.

[Brief Description of the Drawings]

FIG.1 is a diagram showing the construction of a processing vessel of an MOCVD apparatus 100 used in the present invention;

25 FIG.2 is a diagram showing the construction of an MOCVD apparatus 200 according to a first embodiment of the present invention;

FIG.3 is a diagram showing the construction of

an MOCVD apparatus 200 according to a second embodiment of the present invention;

FIG.4 is a diagram showing the construction of an MOCVD apparatus 200 according to a third embodiment
5 of the present invention;

FIG.5 is a diagram showing the construction of an MOCVD apparatus 200 according to a fourth embodiment of the present invention;

FIG.6 is a flowchart showing an example of
10 processing for controlling a source gas concentration in a mixed gas according to any of the first through fourth embodiment; and

FIG.7 is a diagram showing an example of an FTIR spectrum obtained for $W(CO)_6$;

15 [Description of the Reference Numbers]

10 source bottle

12A mass-flow controller (MFC)

12B mass-flow controller (MFC)

14 turbo molecular pump (TMP)

20 16 dry pump (DP)

18 manometer

20 valve

21 valve

23 valve

25 25 valve

26 valve

30 source gas line

31 diluting gas line
32 evacuation line
33 pre-flow line
35 bypass line
5 40 Fourier-transform infrared spectrometer (FTIR)
100 processing vessel
110 showerhead
120 processing vessel body
130 stage
10 140 gate valve
200 MOCVD apparatus

[Name of the Document] Abstract

[Abstract]

[Object] To provide a CVD apparatus, in which the concentration of a source gas supplied to a processing vessel of the CVD apparatus together with a carrier gas for conducting a CVD process is adjusted with high precision and with high speed even when the CVD process is conducted by using a low vapor pressure source material.

10 [Solution Means] A film-formation apparatus, comprising: a film-formation chamber; and a source gas supplying apparatus supplying a source gas to said film-formation chamber together with a carrier gas, said source gas supplying apparatus comprising: a
15 concentration detector detecting a concentration of said source gas; and a gas flow controller controlling a flow rate of an inert gas added to said carrier gas based on a result of measurement of said concentration of said source gas obtained by said concentration detector. It
20 becomes possible to readily and speedily control the source gas concentration by increasing or decreasing the flow rate of an inert gas.

[Selected Figure] Fig. 4

【書類名】 図面 — DRAWINGS

【図 1】 NAME OF DOCUMENT
FIG. 1

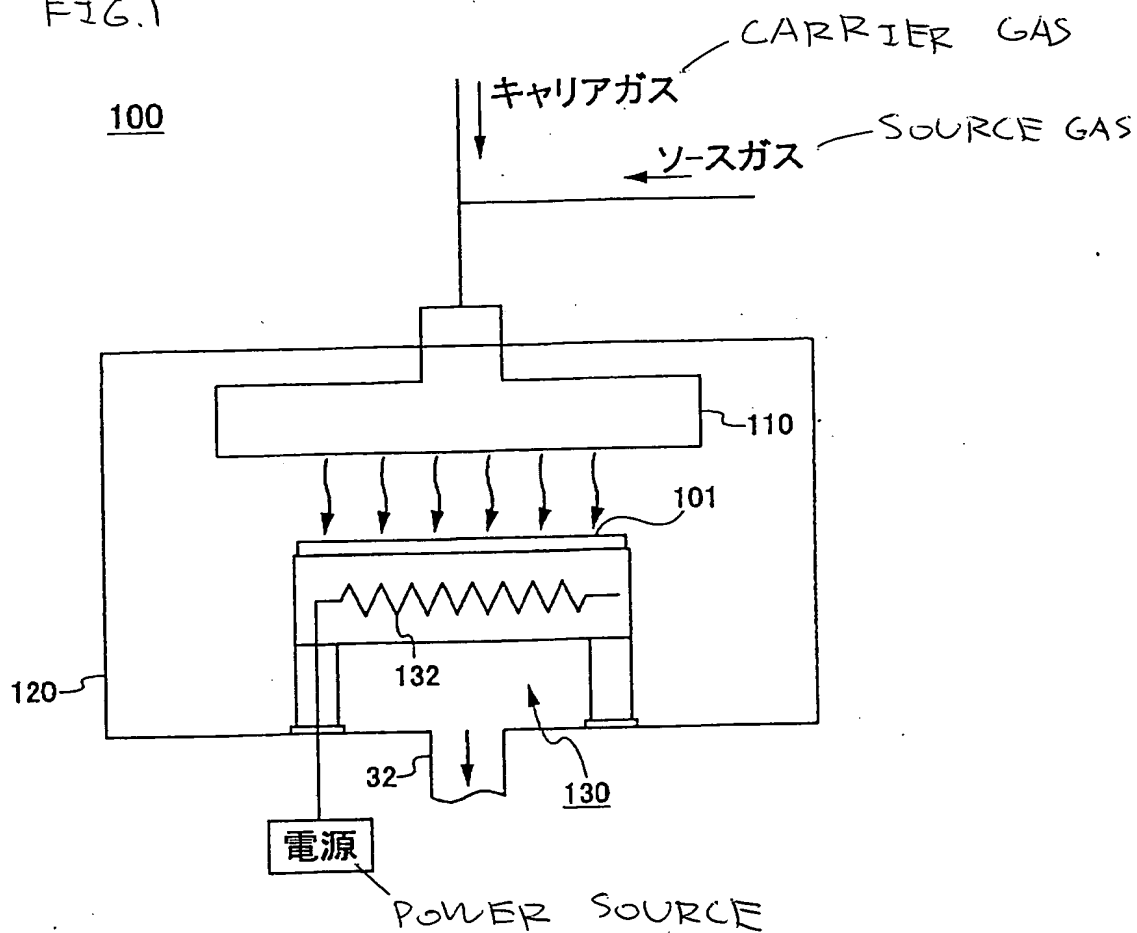
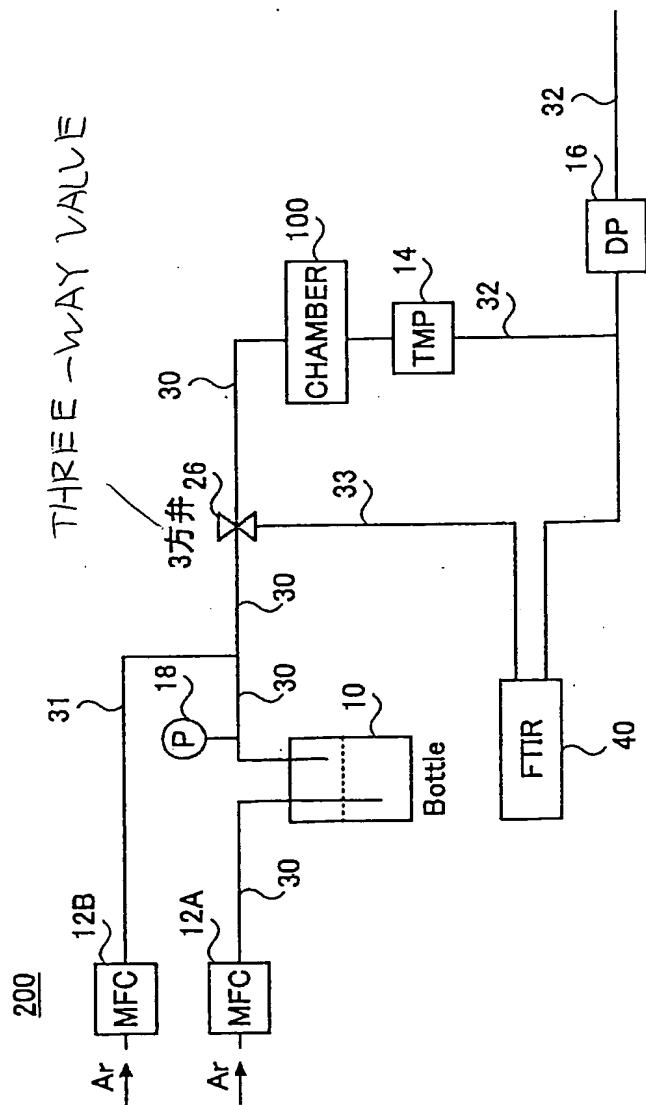
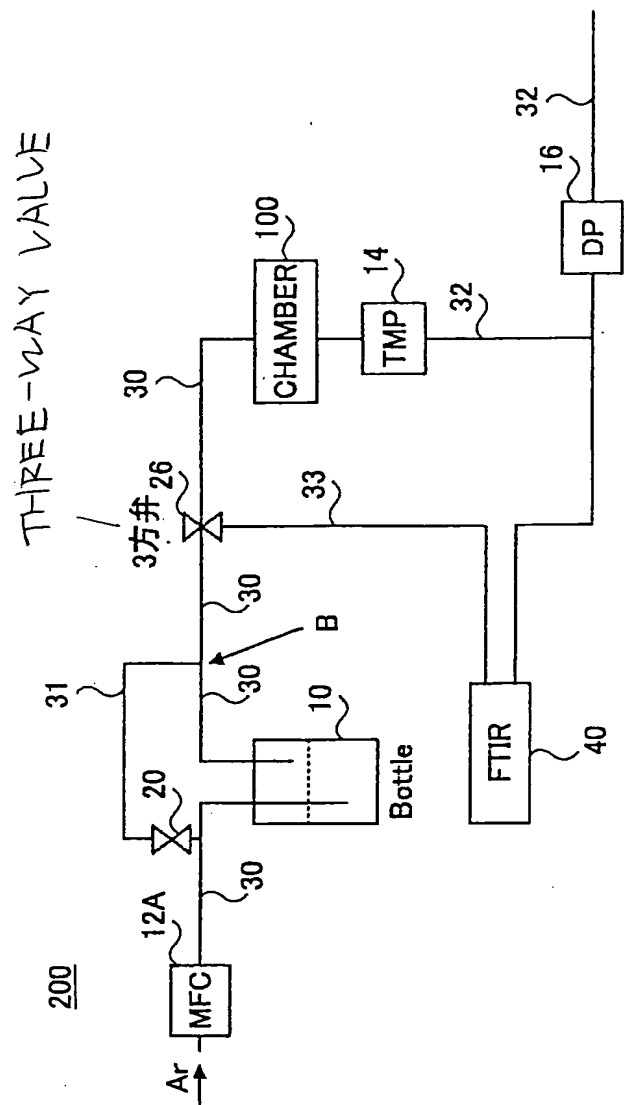


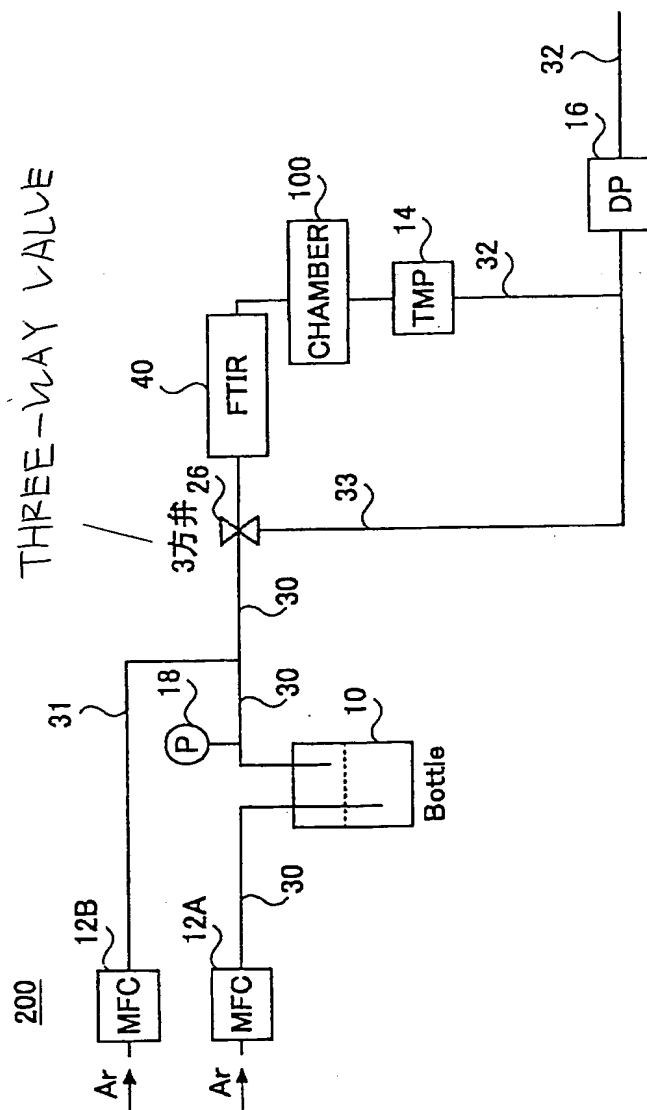
FIG. 2



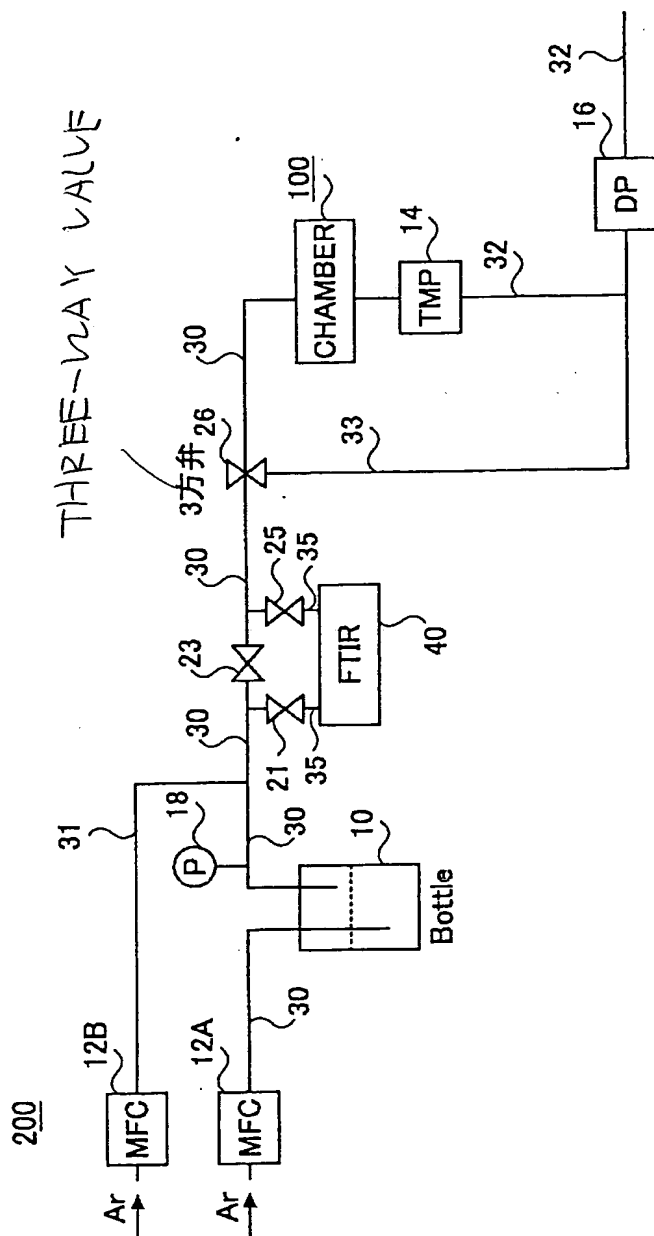
【図3】 FIG.3



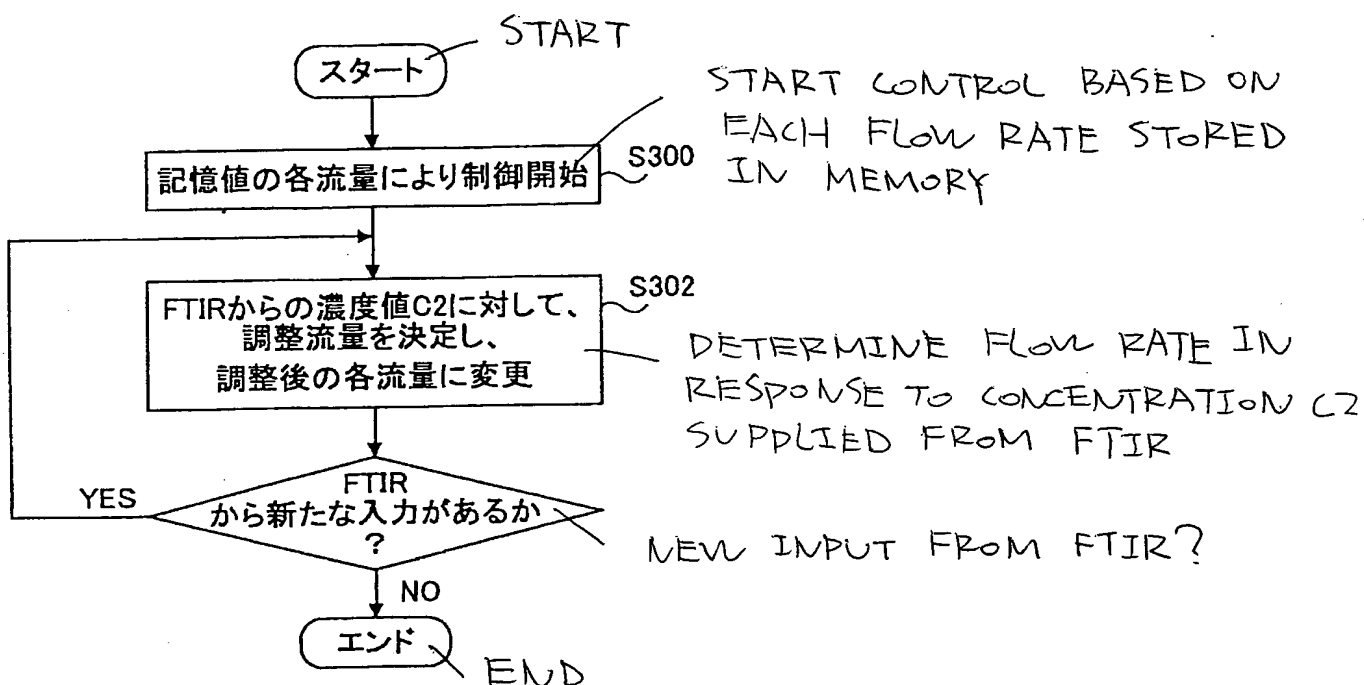
【図4】 FIG.4



【図 5】 FIG. 5



【図 6】 FIG. 6



【図 7】 FIG. 7

